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Scientific and Technical Information Office

# WEAR OF SINGLE-CRYSTAL SILICON CARBIDE IN CONTACT WITH VARIOUS METALS IN VACUUM

by Kazuhisa Miyoshi\* and Donald H. Buckley

#### Lewis Research Center

#### SUMMARY

An investigation was conducted to examine the wear behavior of single-crystal silicon carbide in sliding contact with various metals and the nature of interaction of silicon carbide wear debris. Sliding friction experiments were conducted with a flat silicon carbide on the (0001) basal plane in the  $\langle 10\bar{1}0\rangle$  directions in contact with metal riders of the transition metals (tungsten, iron, rhodium, nickel, titanium, and cobalt), and the metals copper, and aluminum. All experiments were conducted with a load of 30 grams (0.29 N) at a sliding velocity of 3 millimeters per minute, in a vacuum of  $10^{-8}$  pascal, and at  $25^{\circ}$  C).

The results of the investigation indicate that the hexagon-shaped cracking and fracturing of single-crystal silicon carbide occurs during sliding against a metal rider. This cracking and fracturing is believed to be due to cleavage of both prismatic and basal planes. Silicon carbide wear debris, which is produced by brittle fracture during sliding, slides or rolls on both the metal and silicon carbide and produces grooves and indentations on these surfaces. The wear scars of aluminum and titanium, which have much stronger chemical affinity for silicon and carbon, are generally rougher than those of the other metals. Fracturing and cracking along the grain boundary of rhodium and tungsten are observed. These may be primarily due to the greater shear moduli of the metals.

#### INTRODUCTION

Examination by the present authors of single-crystal silicon carbide in sliding contact with various metals revealed that (1) the coefficient of friction for silicon carbide

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is related to the relative chemical activity of these metals, (2) the chemical activity of metal to silicon and carbon and the shear modulus of the metal are important in metal-transfer and the form of the metal wear debris generated (ref. 1), and (3) wear debris of silicon carbide may be produced by brittle fracture during sliding contact with titanium (ref. 2). The examinations of data also indicated no significant changes in coefficient of friction with load. When repeated passes were made of the metal riders over the same single-crystal silicon carbide surface, the coefficient of friction generally decreased with number of passes to an equilibrium value, which seemed to depend on the nature of transfer of the metals (ref. 1). It would be of further interest to know the wear behavior of silicon carbide and metals in sliding contact. The knowledge gained in such studies may lead to a better understanding of wear properties of silicon carbide.

The objective of the present paper is to examine the wear behavior of single-crystal silicon carbide in contact with various metals. The nature of interaction of silicon carbide wear debris was also examined relative to the wear behavior. The metal riders of tungsten, iron, rhodium, nickel, titanium, cobalt, copper, and aluminum were all polycrystalline. All friction experiments were conducted with loads of 30 grams (0.29 N), at a sliding velocity of 3 millimeters per minute, in a vacuum of  $10^{-8}$  pascal, and at  $25^{\circ}$  C, with a flat silicon carbide (0001) basal plane in the  $\langle 10\overline{10} \rangle$  directions.

#### MATERIALS

The single-crystal silicon carbide platelets used in these experiments were a 99.9-percent pure compound of silicon and carbon (ref. 1). The silicon carbide has a hexagonal close-packed crystal structure. The C direction was perpendicular to the sliding interface with the basal plane therefore parallel to the interface.

The metals were all polycrystalline. The titanium was 99.97 percent pure, the copper was 99.999 percent pure, and all the other metals were 99.99 percent pure (ref. 1).

#### EXPERIMENTAL APPARATUS AND PROCEDURE

The apparatus used in the investigation was mounted in an ultrahigh vacuum system. The apparatus is capable of measuring adhesion, load, and friction. The vacuum system also contained tools for surface analysis, an Auger emission spectrometer (AES), and a LEED (low-energy electron diffraction) system (ref. 1).

The disk flat of silicon carbide and metal pin specimens were polished with a diamond powder and with an aluminum oxide  $(Al_2O_3)$  powder. Both powders had particle diameters of 3 and 1 micrometer. The radius of the pin specimens was 0.79 millimeter. The disk and pin surfaces were rinsed with 200-proof ethyl alcohol.

The specimens were placed in the vacuum chamber, and the system was evacuated and subsequently baked out to obtain a pressure of  $10^{-8}$  pascal ( $10^{-10}$  torr). When this pressure was obtained, argon gas was bled back into the vacuum chamber to a pressure of 1.3 pascals. A 1000-volt-direct-current potential was applied, and the specimens (both disk and rider) were argon sputter bombarded for 30 minutes. One hour after the sputtering operation was completed the vacuum chamber was reevacuated, and AES spectra of the disk surface were obtained to determine the degree of surface cleanliness. When the desired degree of cleanliness of disk surface was achieved, friction experiments were conducted (refs. 1 to 3).

Loads of 30 grams (0.29 N) were applied to the pin-disk contact. Both load and friction force were continuously monitored during a friction experiment. Sliding velocity was 3 millimeters per minute, with a total sliding distance of 2.5 millimeters. All friction experiments in vacuum were conducted with the system evacuated to a pressure of  $10^{-8}$  pascal.

#### RESULTS AND DISCUSSION

#### Wear Behavior of Silicon Carbide

The sliding of a metal rider on silicon carbide results in cracks along cleavage planes of  $\{10\overline{1}0\}$ . Figure 1 shows scanning electron micrographs of the wear tracks on silicon carbide surfaces generated by 10 passes of the rhodium and copper riders. The cracks, which are observed in the wear tracks, primarily propagate along cleavage planes of  $\{10\overline{1}0\}$ . In figure 1(a) a hexagon-shaped light area at the beginning of the wear track is a large damaged area where the cracks were generated, propagated, and then intersected during loading and sliding by the rhodium rider. It is anticipated from figure 1(a) that subsurface-cleavage cracking of (0001) planes also occurs. Figure 1(b) indicates the surface cracking, observed on the side of the wear track, along cleavage planes of  $\{10\overline{1}0\}$ .

Figure 2 reveals the wear tracks for silicon carbide contacting titanium characterized by nearly complete hexagon-shaped pit. Wear debris particles (as seen in fig. 1) have been already ejected from the wear track. Again, the fracture is primarily due to the surface cracking of the  $\{10\overline{1}0\}$  planes and the subsurface cracking of the  $\{0001\}$  planes, as anticipated, parallel to the interface. A scanning electron micrograph and an X-ray energy dispersive analysis for titanium on the silicon carbide surface are shown in figure 2. The concentrations of white spots in figure 2(b) correspond to those locations in figure 2(a) where titanium transfer is evident. A large amount of thin titanium film was seen around the hexagon-shaped pit in figure 2(a). These figures indicate that the cohesive bonds on both the silicon carbide and titanium surfaces fractured near the strong adhesive bonds at the interface.

In fact, the dislodged gross wear particle of silicon carbide, which was turned over, has a large amount of titanium on its back as shown in figure 3. Figure 3 shows a scanning electron micrograph and an X-ray energy-dispersive analysis of a silicon carbide wear track.

Figure 4 also reveals the dislodged gross wear debris observed on the sides of wear tracks on silicon carbide surfaces generated by 10 passes of the aluminum rider. Thus, fracture may readily occur in silicon carbide near the adhesive bond, and silicon carbide wear debris particles may be formed.

#### Nature of Interaction on Silicon Carbide Wear Debris

The wear scars on metals after sliding on silicon carbide surfaces may contain the silicon carbide wear debris. Figures 5 to 7 indicate that the wear debris of silicon carbide, produced in sliding on rhodium, copper, and cobalt, transferred to or was embedded in the metal riders. The wear debris generally slides or rolles on the metal surfaces and produces the indentations and deep grooves shown in figures 5 to 7. Figure 6 shows straight grooves, along the sliding direction on the metal, formed by the sliding of silicon carbide wear debris. Figure 7 also shows straight rows of indentations, along the sliding direction, formed by the rolling of silicon carbide wear debris.

On the other hand, figure 8 reveals that silicon carbide wear debris was produced and embedded in the metal by sliding, that it plowed the silicon carbide surface itself, and that the groove is produced in a plastic manner on the surface of silicon carbide. Generally, the silicon carbide wear debris surface is covered with metal film as indicated in the X-ray map of figure 8.

#### Wear Behavior of Metals

The wear scars on the metal riders after sliding against silicon carbide revealed a large number of plastically deformed grooves and indentations (which seem to depend on the nature of transfer of silicon carbide and the metal) formed primarily by the sliding and rolling of silicon carbide wear debris. (See fig. 9.) The less the chemical activity and the greater the resistance to shear of the metal, with the exception of rhodium and tungsten, the less the transfer to silicon carbide (ref. 1). (See tables I and II.) The wear scars are generally rougher with aluminum and titanium, which have much stronger chemical affinity and less resistance to shear than copper, nickel, cobalt, or iron. The wear scar of iron is much smoother, with fewer deep grooves and indentations (fig. 9(f)). The titanium wear scar has the roughest surface (fig. 10). This reflects the large amount of silicon carbide transfer to titanium. Figure 10 reveals that most of the large contact indentations contain silicon carbide wear debris.

Rhodium and tungsten have the greatest resistance to shear of the metals (see tables I and II) and produce and transfer lumps of wear particles (ref. 1). The wear scar on the rhodium rider (fig. 11) reveals evidence of fractured surface damage in addition to plastically deformed grooves and indentations primarily produced by sliding and rolling of silicon carbide wear debris. Further, with rhodium, a significant degree of cracking initiates in a grain boundary and extends along the grain boundaries of rhodium. Such a crack is shown in the micrograph of figure 12.

Figure 13, scanning electron microg aphs of wear scars on the tungsten rider generated by 3 and 10 passes of the tungsten riders, reveals evidence of fractured surface damage and cracking along the grain boundary. In figure 13(a) the tungsten wear debris transferred back to the tungsten. The fracturing and cracking of rhodium and tungsten may be primarily due to the greater shear moduli of rhodium and tungsten.

#### CONCLUSIONS

As a result of observations of the wear behavior of single-crystal silicon carbide and metals in sliding contacts, the following conclusions are drawn:

- (1) Metal riders sliding against single-crystal silicon carbide generate hexagonshaped cracking and fracturing in the silicon carbide. This cracking and fracturing is believed to be due to cleavages of both prismatic and basal planes.
- (2) The silicon carbide wear debris produced by brittle fracture during sliding, slides or rolls on both the metal and silicon carbide and produces grooves and indentations on these surfaces.
- (3) The wear scars of the metals aluminum and titanium, which have much stronger chemical affinity for silicon and carbon, are generally rougher than those of other metals.
- (4) The fracturing and cracking along the grain boundary of rhodium and tungsten observed may be primarily due to the greater shear moduli of these metals.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 29, 1977,
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## TABLE I. - CHARACTERISTICS OF METAL TRANSFER TO SINGLE-CRYSTAL SILICON CARBIDE (0001) SURFACE

[Transferred after 10 passes sliding +; not transferred after 10 passes sliding -; transferred after single pass sliding (+).]

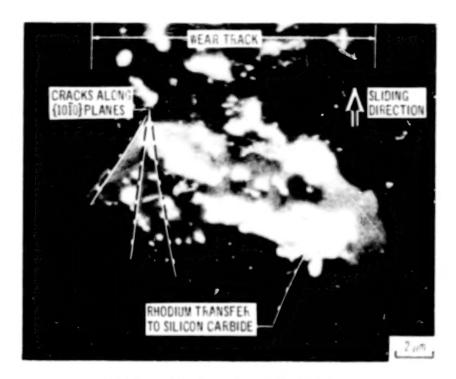
| The form of metal transfer | Al  | Ti  | Cu | Ni | Co | Fe | Rh | w | SiC |
|----------------------------|-----|-----|----|----|----|----|----|---|-----|
| Particle:                  |     |     |    |    |    |    |    |   |     |
| Very small                 | +   | +   | +  | +  | +  | +  | +  | + | +   |
| Piled-up                   | +   | +   | +  | +  | +  | +  | -  | - | -   |
| Thin film:                 |     |     |    |    |    |    |    |   |     |
| Streak                     | +   | +   | +  | +  | +  | -  | -  | - | -   |
| Multilayer (piled-up)      | +   | +   | -  | -  | -  | -  | -  | - | -   |
| Lump                       | (+) | (+) | -  | -  | -  | -  | +  | + | +   |

a<sub>Ref. 2.</sub>

TABLE II. - PHYSICAL PROPERTIES

| Metal | Shea | r modulus<br>(a)       | Cohesive energy (a) |             |  |  |
|-------|------|------------------------|---------------------|-------------|--|--|
|       | mPa  | kg/cm <sup>2</sup>     | kJ/g-atom           | kcal/g-atom |  |  |
| Al    | 26.6 | 0.271×10 <sup>-6</sup> | 322                 | 76.9        |  |  |
| Ti    | 39,3 | .401                   | 469                 | 112,2       |  |  |
| Cu    | 45.1 | .460                   | 338                 | 80.8        |  |  |
| Ni    | 75.0 | .765                   | 428                 | 102,3       |  |  |
| Co    | 76.4 | .779                   | 426                 | 101.7       |  |  |
| Fe    | 81.5 | .831                   | 416                 | 99.4        |  |  |
| Rh    | 147  | 1,50                   | 556                 | 133.0       |  |  |
| w     | 153  | 1.56                   | 835.5               | 199.7       |  |  |
| SIC   | 200  | 2.04                   | 523                 | 125         |  |  |

<sup>&</sup>lt;sup>a</sup>Data from ref. 4 for metals and refs. 5 and 6 for SiC.

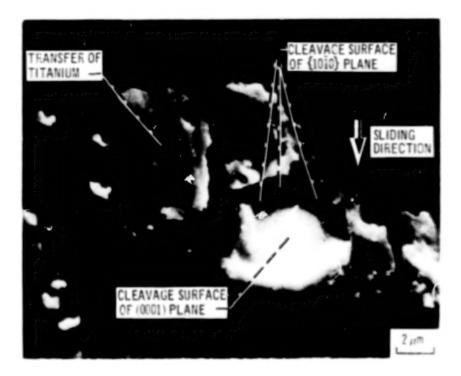


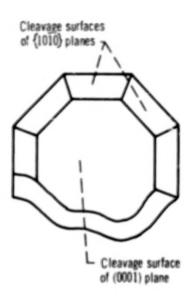
(a) Silicon carbide after contact with rhodium rider.

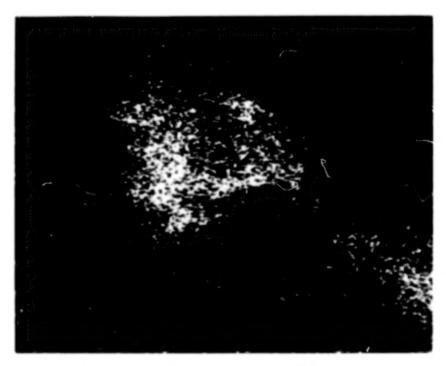


(b) Silicon carbide after contact with copper rider.

Figure 1. - Cracking of silicon carbide as result of sliding contact with metal riders. Ten passes on SiC (0001) surface; sliding direction, <1010>; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure, 10<sup>-8</sup> Pa. (Scanning electron micrographs.)

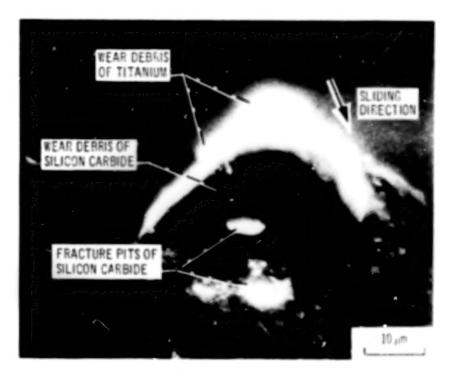






(b) Titanium  $K_{\rm cz}$  X-ray of silicon carbide; 1.5x10 $^4$  counts.

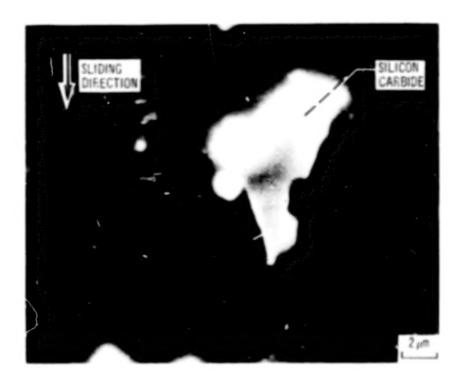
Figure 2. - Hexagon shaped pit of silicon carbide as result of sliding contact with titanium rider. Ten passes on SiC (0001) surface; sliding direction, <10\overline{10\overl





(b) Titanium  $\rm K_{\rm x}$  X-ray map of silicon carbide surface; 1.5x10  $^{\rm 4}$  counts.

Figure 3. - Titanium and silicon carbide wear debris on SiC surface produced during sliding contact. Ten passes of Ti rider on SiC (0001) surface; sliding direction, <10 $\overline{10}$  >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.



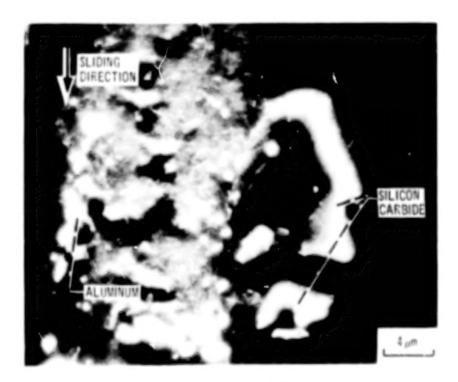
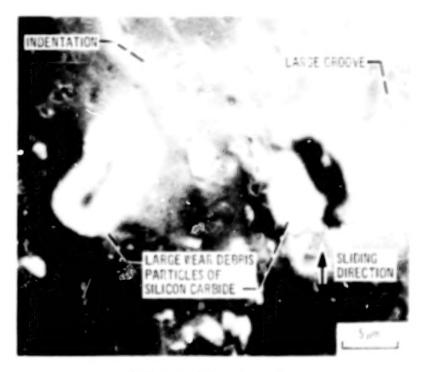
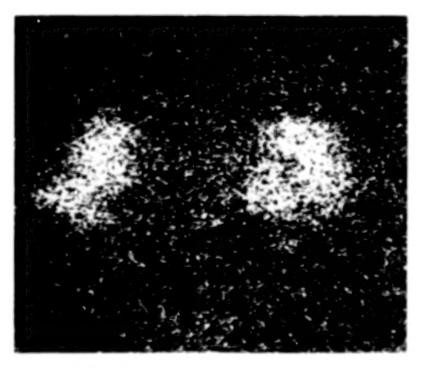


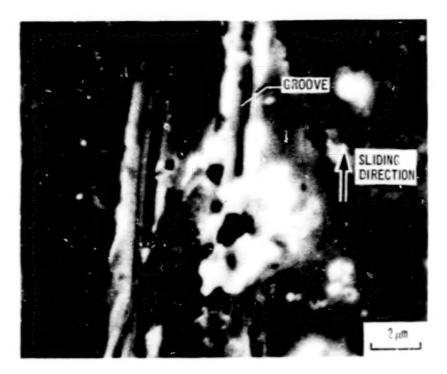
Figure 4. - Large wear debris of silicon carbide on SiC surface produced during sliding contact with aluminum rider. Ten passes on SiC (0001) surface; sliding direction, <10 $\overline{10}$ , sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa. (Scanning electron micrographs.)





(b) Sificon  $\mbox{K}_{tz}$  X-ray map of rhodium rider;  $2.0 \mbox{x} 10^4$  counts.

Figure 5. - Grooves and indentations on rhodium rider produced by silicon carbide wear debris during sliding contact. Ten passes on SiC (0001) surface; sliding direction, <1010>; sliding velocity, 3 mm/min; load, 30 g (0.29 N), temperature, 25°C; vacuum pressure,  $10^{-8}$  Pa.



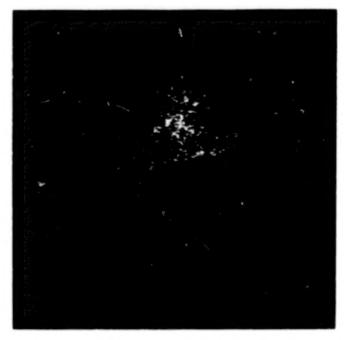


(b) Silicon  $\mbox{K}_{\alpha}$  X-ray map of copper rider;  $1.5 \mbox{x} 10^4$  counts.

Figure 6. - Grooves on copper rider produced by scratching of silicon carbide wear debris during sliding contact. Ten passes on SiC (0001) surface; sliding direction, <10 $\bar{1}0$ ; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25°C; vacuum pressure,  $10^{-8}$  Pa.

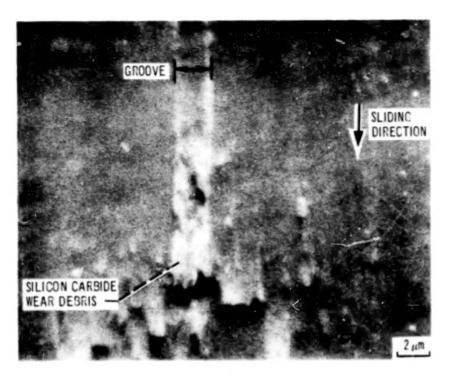


(a) Scanning electron micrograph.



(b) Silicon  $K_{zz}$  X-ray map of cobalt rider,  $1.0 x 10^4$  counts.

Figure 7. - Indentations on cobalt rider produced by rolling of silicon carbide wear debris during sliding contact. Ten passes on SiC (0001) surface; sliding direction,  $<10\bar{1}0>$ ; sliding welocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.

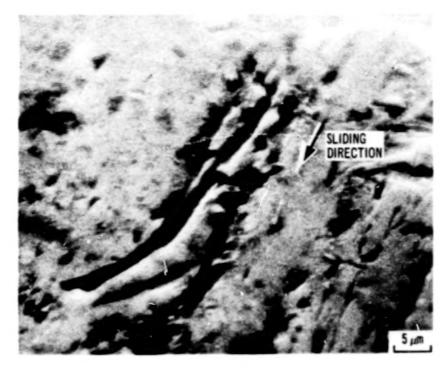


(a) Scanning electron micrograph.

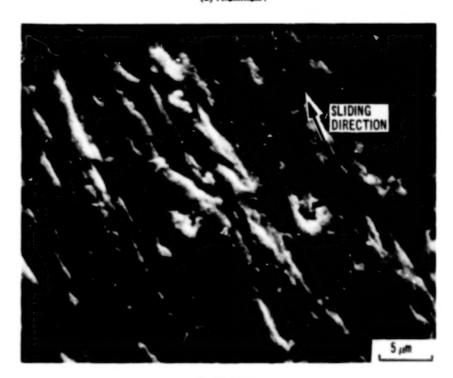


(b) Titanium  $K_{\alpha}$  X-ray map of silicon carbide surface, 1.7x10<sup>4</sup> counts.

Figure 8. - Groove on silicon carbide produced by silicon carbide wear debris during sliding contact with titanium rider. Ten passes on SiC (0001) surface; sliding direction, <10 $\overline{10}$  >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.

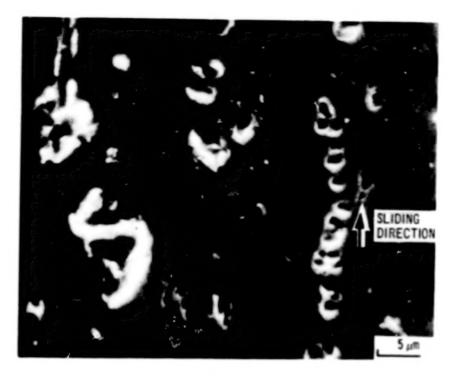


(a) Aluminum.

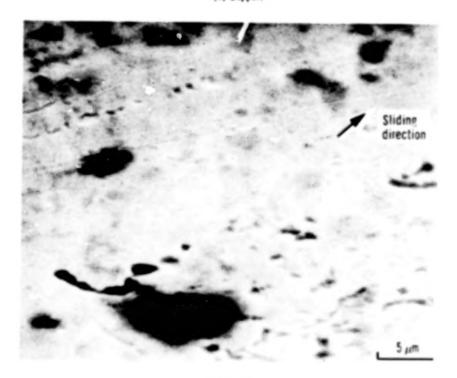


(b) Titanium.

Figure 9. - Wear scars on metal riders produced during sliding contact with silicon carbide. Ten passes on SiC (0001) surface; sliding direction, <10 $\overline{10}$ >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa. (Scanning electron micrographs.)

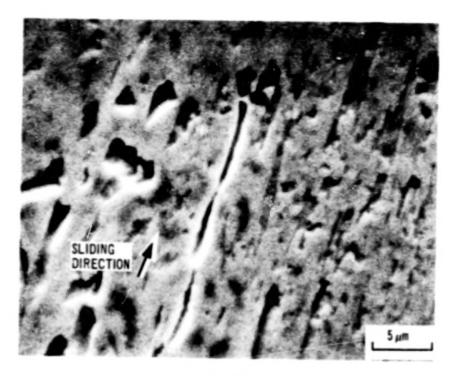


(c) Copper.

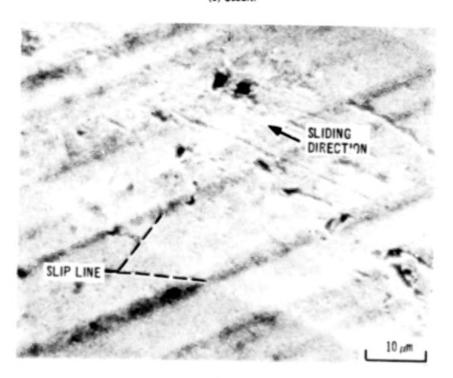


(d) Nickel.

Figure 9. - Continued.

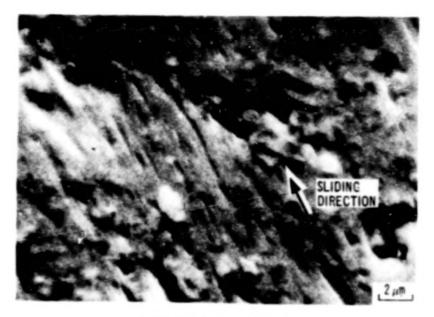


(e) Cobalt.



(f) Iron.

Figure 9. - Concluded.

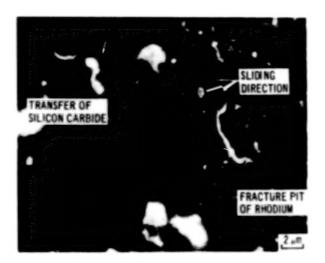


(a) Scanning electron micrograph.



(b) Silicon  $\mbox{K}_{z}$  X-ray map of titanium rider;  $3.0 \mbox{x} 10^4$  counts.

Figure 10. - Rough surface of titanium rider showing large amount of silicon carbide transfer. Ten passes on SiC (0001) surface; sliding direction, <10 $\overline{10}$ >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.





(a) Scanning electron micrograph and silicon K<sub>et</sub> X-ray map of rhodium rider; 1.5x10<sup>4</sup> coests.



(b) Scanning electron micrograph (high magnification).

Figure 11. - Wear scar on rhodium rider showing grooves, indentations, and fracture. Ten passes on SiC (0001) surface; sliding direction, <1010 >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.

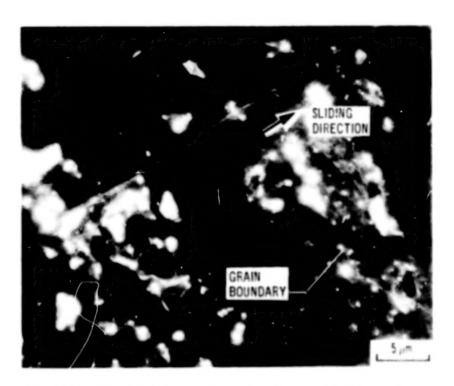
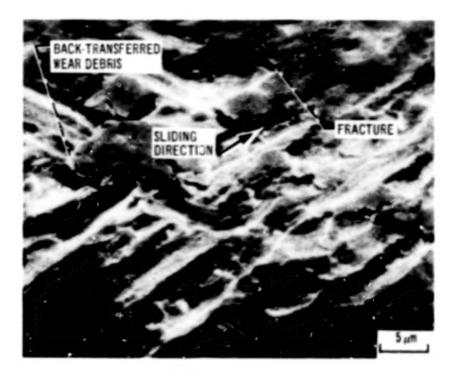
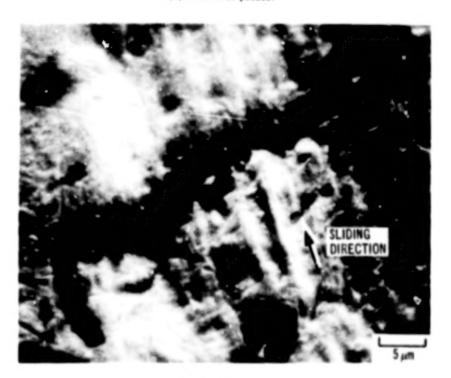


Figure 12. Wear scar on rhodium rider showing cracks propagating along grain boundary. Three passes on SiC (0001) surface; sliding direction, <1010>; sliding velocity, 3 mm/min; load, 30 g (0.29 N) temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.



(a) After three passes.



(b) After 10 passes.

Figure 13. - Tungsten rider wear scars produced during sliding contact with silicon carbide (0001) surface. Sliding direction, <10 $\bar{1}0$ >; sliding velocity, 3 mm/min; load, 30 g (0.29 N); temperature, 25° C; vacuum pressure,  $10^{-8}$  Pa.

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| 16.                                    | Abstract  |   |                                       |                            |                   |  |  |  |
|  | Sliding friction experiments we   | ere conducted in                        | vacuum with single-                   | crystal silicon            | carbide (0001)    |  |  |  |
|  | surface in contact with transiti  | on metals (tungs                        | ten, iron, rhodium,                   | nickel, titanium           | , and cobalt),    |  |  |  |
|  | copper, and aluminum. The he  | exagon-shaped co                        | acking and fracturi                   | ng of silicon car          | rbide that oc-    |  |  |  |
| 1                                      | curred is believed to be due to   |   |                                       | -                          |                   |  |  |  |
|  |   |   | -                                     |                            |                   |  |  |  |
|  | carbide wear debris, which wa   |   |                                       |                            |                   |  |  |  |
| 1                                      | and silicon carbide and produce   | es grooves and in                       | dentations on these                   | surfaces. The              | wear scars        |  |  |  |
|  | of aluminum and titanium, which   | ch have much str                        | onger chemical affi                   | nity for silicon :         | and carbon,       |  |  |  |
|  | are generally rougher than those  | se of the other m                       | etals. Fracturing                     | and cracking alo           | ng the grain      |  |  |  |
|  |   |   |                                       | _                          |                   |  |  |  |
|  | boundary of rhodium and tungsten were observed. These may be primarily due to the greater shear moduli of the metals.   |   |                                       |                            |                   |  |  |  |
|  | shear moduli of the metals.   |   |                                       |                            |                   |  |  |  |
|  |   |   |                                       |                            |                   |  |  |  |
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|  |   |   |                                       |                            |                   |  |  |  |
|  |   |   |                                       |                            |                   |  |  |  |
| 17.                                    | Key Words (Suggested by Author(s))  | by Author(s) 18. Distribution Statement |                                       |                            |                   |  |  |  |
|  | Single-crystal silicon carbide;   | Metals;                                 | Unclassified - unlimited              |                            |                   |  |  |  |
|  | Auger; Wear; Fracture   |   | STAR Category 27                      |                            |                   |  |  |  |
|  |   | ,,                                      |                                       | Cini Category at           |                   |  |  |  |
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